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Investigation of plumbum-containing slags and ways for their reclaiming

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Abstract. The paper deals with analysis and findings of investigation of technogenic plumbum-zinc slag residuals that involve a great deal of poisonous compounds such as cadmium, osmium, plumbum, zinc, hazardous origins of environmental contamination. In connection with the outdoor storing of slags, an excess of the plumbum critical concentration was derived. Technogenic slag residuals' reclamation is crucial for minimizing the detrimental effect on health and safety, improving the region's ecological setting. At that, slags are high-value crude materials involving rare-earth and non-ferrous metal compunds. The paper demonstrates the findings of laboratory tests of slags to specify the quantitative and qualitative composition of high-value components in the plumbum residuals, the ability of their subsequent treatment and reclamation. The heavy slag fraction material was investigated on JEOL IXA-8230 Electron Probe microanalyzer. The running slag sample's X-ray diffraction analysis was carried out on DRON-4 diffractometer with graphite monochromator, Cu radiation. Heavy fractions were separated from the samples, artifactural polished microsections (briquets) were made. The polished microsections were investigated in index-matching fluids and under LEICA DM 2500P microscope. The findings identified that plumbum slags involve a considerable body of non-ferrous metal compounds, which allow to make the reclamation of poisonous plumbum residuals functional and cost-effective.

Keywords: *plumbum-containing slags, heavy metal, high-value components, slag, reclamation.*

1. Introduction

In metallurgy, the essential environmental conservation area is to implement disposable technologies and crude materials' multiple use technologies. This provides ore benefication, rational completeness of major and accompanying elements' extraction, residuals' reclamation without harming the environment.

In 2011, due to various economic reasons, Shymkent Plumbum Factory was decisively closed. Nevertheless, the environmental situation of the city is as before negatively exerted by collected slag residuals to the number of 2 million tons kept in outdoor storings under the action of atmospheric precipitates, aerial oxygen and sun rays [1]. The plumbum factory slags are important crude materials containing various non-ferrous metals, currently non-ferrous metals obtained from secondary crude materials play an important role in overall balance of non-ferrous metals' production and consumption in Kazakhstan: their share with regard to the total non-ferrous metal production is about 25% [2]. For instance, it is the authors' opinion that plumbum has an appreciated economic value, plumbum application area has changed in recent years, now approximately 80% of world usage is in the electric battery sector. Plumbum's pliability, density and anti-corrosion properties till now are aggressively applied in construction of reservoirs for caustic liquids' storing and defense against radiation and X-rays. Plumbum is applied when producing pigments, paints, other chemical compounds [3]. Automobiles use plumbum-acid batteries. The cause for common plumbum use in batteries in automobile and industrial engineering is that plumbum can provide a great deal of electricity needed to power a car starter in a short period of time [4].

Besides, batteries are applied for providing the electrical current, for driving such massive carriers as electric or diesel submarines and locomotives, as a source for reserve energy in plants involving critical functions such as telecommunication and inpatient facilities. Plumbum has the highest use factor in metal reworking. It is applied in IT sector as additive and solder, in chemical industry as a component in paints and reagents' production [5]. By virtue of its unique physico-chemical features, plumbum has identified a spot in different engineering products' manufacture, for instance in protective roofs of structures and buildings. The metal's high noncorrodibility, strength, simplicity of operation are major benefits in its application, as well as for application in medical goods to protect against gamma rays, in spectrographic and X-ray equipment production. Plumbum is a constituent of printing alloys, anti-friction metals, yellow metals, bronzes [6].

One more non-ferrous metal offered for recovery when treating plumbum residues is zinc. It is applied to galvanize metal goods for giving them corrosion-inhibiting properties. Thereby, the quest for it persists strong owing to the explosion in the rust-inhibiting coatings' production. Zinc is also applied in typesetting materials, alloys (silver, nickel, brass) production [7]. Zinc compounds are also applied when producing glaze, glass, rubber pigments for paints. Another substantial range of use is for formulating corrective facial pastes, pharmaceutical dosages [8].

Hereby, the most profit-proved and powerful way for cutting down spoils' detrimental effect on the city's ecological setting is their subsequent treatment. In fact, after non-ferrous metals' recovery, it is possible to apply slags when producing constructional materials and cement, as plumbum residuals consist of 75-85% of silicon, calcium and iron oxides [9, 10].

2. Materials and methods

2.1. Experimental part

The research thing is plumbum-containing slag dumps from the plumbum factory, that are inventoriable costs. For identification of plumbum slags' reclamation and treatment ways to recover plumbum and zinc oxides, there are many papers founded on the necessity for identification of non-ferrous metals and other compounds' quantitative content and chemical composition [11]. The main research objective is development of enabling technology to treat plumbum residuals allowing to involve them in reclamation in the capacity of recoverable resources. This in turn will enable reasonably apply environmental assets and decrease the areas with the residuals [12].

Tentative data on the plumbum residuals received in the operating cycle demonstrated that the plumbum residuals are metallurgical slags. The plumbum-containing slag particles have the shape of irregular granules, the material density in the granular material is 2 t/m3, the slope of repose is about 35°, the particle size essentially ranges from 2 to 6 mm, at that they represent a little of particles with the size of around 10 mm. Table 1 represents dry plumbum residual components [13].

Table 1. Dry plumbum-zinc residual components

Element	Pb	Zn	Cu	Fe	SiO_2	CaO	K_2O	S	O
Digital quantity	2.38	9.81	0.97	25.31	24.62	16.21	1.42	1.35	10.16

Notice: The digital quantity of the sample is the mean of a randomly chosen sample

To identify the plumbum residuals' chemical composition, the authors made chemical, thermal, X-ray phase and spectral analyses in Almaty at Institute of Geological Sciences named after K.I. Satpayev and at Institute of Metallurgy and Ore Beneficiation of the National Academy of Sciences of the Republic of Kazakhstan [14].

The material constitution was investigated on a black granular slag material with the size of the particle from 2 to 6 mm. The sample was separated into heavy fractions from which artifactural polished microsections (briquets) were made. They were investigated using LEICA DM 2500P microscope. With it, the sample was investigated using the microscope in index-matching fluids, whereby the sample was chosen for subsequent investigations [15, 16].

The running slag sample's X-ray diffraction analysis was carried out on DRON-4 diffractometer with graphite monochromator, Cu radiation. The diffraction patterns' logging characteristics: magnification: 2000 imp; I=20mA; U=35kV; response time: 2 s; shooting theta: 2; detector: 2 deg/min. Semiquantitative analysis was made in terms of the powder sample diffraction patterns applying artificial combinations and similar masses' method. The crystal line phases' quantitative relationships were identified. ASTM Powder diffraction file and clean diffraction patterns were applied to interpret the diffraction patterns. The contents were figured out for the main phases [17]. Table 2 represents probable impurities which characterization cannot be identical because of low contents and availability of only 1-2 diffraction reflections or defective crystallization.

Table 2. The findings of the technological slag sample's semi-quantitative atomic emission spectral analysis

Elements	Elements' content, %	Elements	Elements' content, %
Gold	<0.0002	Silver	0.001
Silicon	>>1.0	Magnesium	>1.0
Aluminum	>1.0	Calcium	>1.0
Copper	0.3	Rhenium	<0.0003
Nickel	0.0025	Chromium	0.015
Antimony	<0.002	Cobalt	0.005
Arsenic	<0.01	Molybdenum	0.01
Iron	>>1.0	Strontium	0.1
Manganese	0.2	Tellurium	<0.003
Titanium	0.3	Lanthanum	0.002
Zinc	>1.0	Bismuth	0.0005
Potassium	<1.0	Beryllium	0.0003
Natrium	>1.0	Zircon	0.01
Tin	0.001	Ytterbium	0.0002
Barium	0.3	Yttrium	0.003
Scandium	0.0005	Antimony	0.07
Vanadium	0.007	Cerium	0.005
Wolfram	0.005	Gallium	0.002
Germanium	0.001	Thallium	<0.0005
Cadmium	<0.0005	Plumbum	0.1
Iridium	<0.001	Niobium	<0.001
Arsenic	<0.01	Mercury	<0,003
Platinum	<0.001	Palladium	<0.0002
Rhodium	<0.0005	Ruthenium	<0.001

3. Results and discussion

Pursuant to the X-ray diffraction analysis data from Table 2, the slag samples are posed by amorphous phases similar in composition to natural origin crystalline phases, specifically iron oxide, zinc oxide, wollastonite, and fayalite. In the immersion preparation in incident light under the microscope, all these phases are outwardly black and amorphous, but no crystalline formations are evidenced [18]. Diffractogram in Figure 1 exhibits mineral phases' identification pursuant to the X-ray diffraction analysis data.

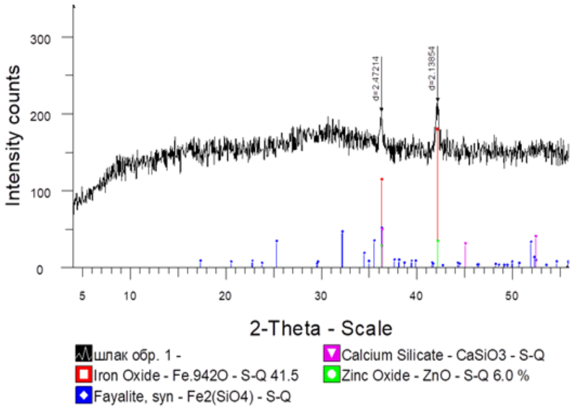

Figure 1. The slag sample diffractogram

Table 3 represents the slag sample phase constitution and interplane distances' findings. All posed diffraction peaks specified in Table 3 appertain only to the mentioned above phases. The typical diffraction reflections are seen, making it possible to determine the phases available.

Table 3. The slag sample phase constitution and interplane distances

d, Å	I %	Phase
2.47214	93.8	Iron Oxide, Fayalite, Zinc Oxide, Calcium Silicate
2.13854	100.0	Iron Oxide, Zinc Oxide

Table 4 represents the crystalline phases' semiquantitative X-ray phase analysis findings [19].

Table 4. The crystalline phases' semiquantitative X-ray phase analysis findings

Mineral phase	Chemical formula	Content, %
Iron oxide	$Fe_{0.942}O$	41.5
Fayalite, syn	$Fe_2(SiO_4)$	35.4
Calcium silicate	$CaSiO_3$	17.2
Zinc oxide	ZnO	6.0

When analyzing Table 3, it was found that the basis of the slag sample is an amorphous substance with the stated crystalline phases with applied reflections.

The investigation of the sample in the polished briquet in the incident light, exhibited in Figure 1, identified that the slag sample is composed of an amorphous matrix with multiple heterogeneous copper mineral phase inclusions outwardly resembling natural copper sulfide minerals, like native copper and chalcopyrite, that are frequently round and isometric, have typical of chalcopyrite light-yellow color [14, 19].

The heavy slag fraction material was investigated on Electron Probe microanalyzer for determination of industrially valuable slag minerals. To that end, the polished briquet surface was thoroughly scanned, this helped to find out in the sample: copper mineral phases, associated artificial plumbum-zinc mineral formations, to investigate their compositions. The investigations were pursued using modern JEOL IXA–8230 Electron Probe microanalyzer.

The polished briquet plane's electron probe scan was made for investigation of the sample matrix and very small inclusions' composition (Figures 2, 3, 4).

Currently, existing methods of plumbum-containing slags' reclamation are economically unsound. One of the essential practical values is to enhance and generate advanced competing technological processes and devices for secondary and technogenic crude materials' treatment.

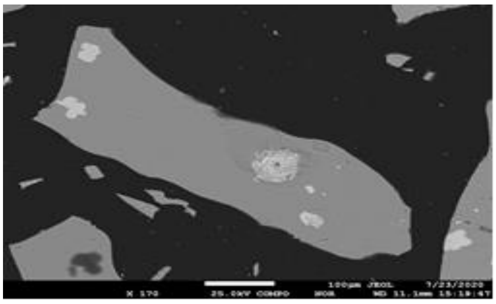

Figure 2. Mineral matrix with copper mineral phases' inclusions

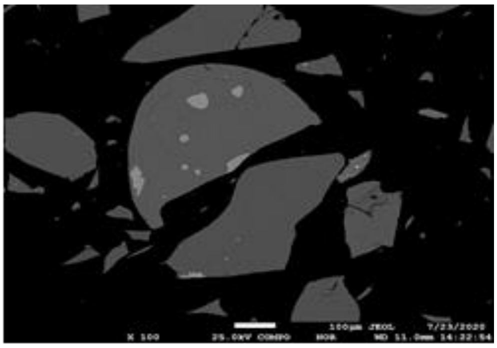

Figure 3. Complex copper mineral phases' electron-probe inclusions in the matrix

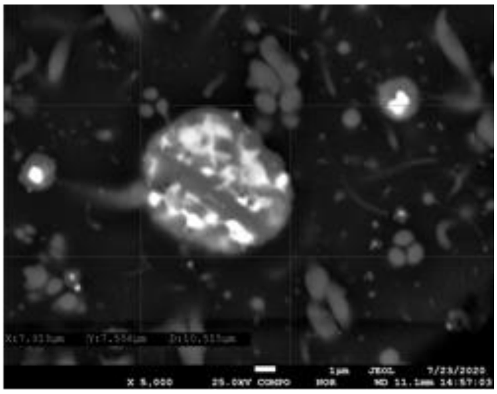

Figure 4. Matrix with complex plumbum-zinc-ferruginous-copper mineral phase oxides inclusions

Comparing the European patent of Carlos Alvarez Carreno and other authors [20], who formulated a way for optional and innocuous to the environment co-extraction of plumbum and silver in the form of a concentrate from hydrometallurgical wastes, it was found that it comprises oxidating desalination hydrometallurgical wastes involving at any rate plumbum and silver with an oxidizer and a chloride solution to guarantee plumbum and silver dissolution selectability by changing them to the state of dissoluble chlorides; and realizing the reaction of an impregnated leach solution received by the above oxidating desalination with a carbonate compound as a settler for the co-deposition of plumbum and silver in the form of a carbonate concentrate [21]. Further, the finished concentrate is purified by contact with a solution of a carbonate compound involving 0.01-0.5% plumbum and silver chlorides in the carbonate concentrate, calculated on a dry weight. The technical result is synchronous plumbum and silver release into a premium concentrate [22].

Based on the above, it can be concluded that pyrometallurgical ways and technics for plumbum and its alloys production from secondary crude materials and technogenic wastes are essentially applied in world practice. This crude material contains plumbum in the form of sulfidoids, sulfates, oxides and other compounds. Plumbum-containing crude materials' reclaiming into intended product is concerned with considerable material expenses demanding the disposal of poisonous plumbum compounds' gas and dust emissions into the atmosphere that leads to the necessity to introduce enhanced ways for the disposal of complex crude materials of plumbum production.

4. Conclusions

The chemical, electron-probe, X-ray phase, spectral analyses' findings identified that the plumbum residuals have considerably great number of non-ferrous metal compounds: up to 1.25% of copper oxide, up to 17% ot zinc oxide, up to 2% of plumbum oxide of the total sample mass. The non-ferrous metals' qualitative composition and content in the plumbum slags makes it possible to make the reclamation of poisonous plumbum residuals functional and cost-effective.

The next advantageous components were detected in the heavy slag sample fraction scanning on the electron probe microanalyzer: copper in terms of sulfides, complex compounds of copper, iron, zinc and plumbum oxides that are met as inclusions in a complex composition amorphous host matrix.

The trial test findings helped us to single out a technology for plumbum and zinc oxides' more complete and selective recovery from the plumbum residuals. When applying the selective way for recovering non-ferrous metals, it is anticipated that the environment's ecological state will be improved, the negative impact on the human health will be decreased owing to the reclamation of poisonous plumbum residuals. At that, a noticeable contribution will be made to the development of the natural and secondary resources' rational use system.

Thereby, the most powerful way for cutting down spoils' detrimental effect on the city's ecological setting is their subsequent treatment with an eye to gain target products popular on the market. After non-ferrous metals' recovery, it will be possible to apply slags when producing constructional materials and cement, when installing paving slabs.

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Құрамында қорғасын бар қождарды зерттеу және оларды кәдеге жаратудың келешегі

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Аңдатпа. Мақалада қорғасын өндірісінің техногенді қож қалдықтарын ғылыми зерттеу нәтижелері мен талдауы берілген. Зауыт жабылғаннан және жойылғаннан кейін қалған қалдықтар қож түрінде топырақтың, жер асты суларының және ауаның экологиялық ластану көзі болып табылады. Қорғасын өндірісінің шлактарында экологиялық ластанудың қауіпті көзі болып табылатын қорғасын, мырыш, осмий, кадмий сияқты көптеген улы қосылыстар бар. Техногенді қож қалдықтарын пайдаға асыру аймақтың экологиялық жағдайын жақсарту және өмір тіршілігінің қауіпсіздігіне теріс әсерін төмендету үшін үлкен маңызға ие. Сонымен қатар, шлактар түсті және сирек кездесетін металдардың қосылыстары бар құнды шикізат болып табылады. Мақалада қорғасын өндірісінің қалдықтарындағы құнды компоненттердің сапалық және сандық құрамын анықтау бойынша қождарды зертханалық зерттеу нәтижелері және оларды әрі қарай өңдеу мен кәдеге жарату мүмкіндігі көрсетілген. JEOL IXA-8230 Electron Probe microanalyzer маркалы электронды–зондты микроанализаторда қождың ауыр фракциясының материалын зерттеу жүргізілді. Қождың орташа сынамасының рентгенодифрактометриялық талдауы cu-сәулеленуі бар ДРОН –4 дифрактометрінде, графитті монохроматорда орындалған. Сынамалардан ауыр фракциялар бөлініп, жылтыратылған жасанды аншлифтер (брикеттер) жасалды. Аншлифтер LEICA DM 2500p маркасының микроскопымен және иммерсиялық сұйықтықтармен зерттелді. Зерттеу нәтижелері бойынша қорғасын қождарында түсті металдар қосылыстарының жеткілікті жоғары мөлшері бар екендігі анықталды: қорғасын оксиді 0.7%-ға дейін және мырыш оксиді қождың салмақтық мөлшерінің 8.5%-на дейін, бұл қорғасын өндірісінің улы қалдықтарын кәдеге жарату процесін техникалық және экономикалық тұрғыдан орынды етуге мүмкіндік береді.

Негізгі сөздер: *құрамында қорғасын бар қождар, ауыр металл, құнды компоненттер, қож, қайта өңдеу.*

Исследования свинецсодержащих шлаков и перпективы их утилизации

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Аннотация. В статье представлены анализ и результаты научных исследований техногенных шлаковых отходов свинцового производства. После закрытия и ликвидации завода, оставшиеся отходы, в виде шлаков, являются источником экологического загрязнения почвы, грунтовых вод и воздуха. Шлаки свинцового производства содержат большое количество токсичных соединений, таких как свинец, цинк, осмий, кадмий, которые являются опасными источниками экологического загрязнения. Утилизация техногенных шлаковых отходов имеет большое значение для снижения отрицательного влияния на безопасность жизнедеятельности и улучшения экологической обстановки региона. В то же время шлаки являются ценным сырьем, содержащим соединения цветных и редкоземельных металлов. В статье показаны результаты лабораторных исследований шлаков по определению качественного и количественного состава ценных компонентов в отходах свинцового производства и возможности дальнейшей их переработки и утилизации. Были проведены исследования материала тяжелой фракции шлака на электронно-зондовом микроанализаторе марки JEOL IXA–8230 Electron Probe microanalyzer. Рентгенодифрактометрический анализ средней пробы шлака выполнен на дифрактометре ДРОН-4 с Cu –излучением, графитовый монохроматор. Из проб были выделены тяжелые фракции и изготовлены полированные искусственные аншлифы (брикеты). Аншлифы изучались под микроскопом марки LEICA DM 2500P и в иммерсионных жидкостях. По результатам исследований выявлено, что в свинцовых шлаках содержится достаточно высокое количество соединений цветных металлов: оксида свинца до 0.7 % и оксида цинка до 8.5 % от весового количества шлака, что позволяет сделать процесс утилизации токсичных отходов свинцового производства технически и экономически целесообразным.

Ключевые слова: *свинцосодержащие шлаки, тяжелые металлы, ценные компоненты, шлак, переработка*.

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